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## (54) Laminated film or sheet and box-shaped vessel prepared therefrom

Verbundfolie oder Verbundplatte und daraus hergestellter kastenförmiger Behälter

Film ou feuille multicouche et récipient en forme de boîte fabriqué à partir de celui-ci

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**Description**

The present invention relates to a laminate film or sheet comprising a layer of a specific resin, a layer of a specified polymer and a paper layer, to a box-shaped vessel (hereinafter referred to as "carton") prepared therefrom, and to processes for the preparation of the film or sheet and vessel.

Recently, cooking methods using an electronic oven have been rapidly introduced. A variety of foods such as dishes and cakes can be easily prepared by using an electronic oven.

When a food is cooked by an electronic oven, the food to be cooked is contained in a tray-shaped vessel formed of a laminate comprising, for example, an inner layer of a 4-methyl-1-pentene polymer and an outer paper layer, and the food is subjected to high-frequency heating.

Foods to be cooked include foods containing oil and fat components such as butter and margarine, for example, pound cakes, and foods on which a sauce containing oil and fat components is poured to complete cooking after heating. It sometimes happens that after heating in an electronic oven or after pouring of a sauce containing oil and fat components after side heating, foods are preserved in a box-shaped vessel.

Since the heretobefore used covering layer of a 4-methyl-1-pentene polymer has a high rigidity, fine cracks are sometimes formed in the covering layer. Moreover, when oil and fat components as described above are present, they permeate into the paper layer constituting the vessel over time, the paper layer as a whole becomes oily thus degrading the appearance of the vessel, and there is a risk of contamination of the hand or clothing with oil and fat components during handling.

The present invention provides a laminate film or sheet having at least three layers, comprising a layer of poly-4-methyl-1-pentene, an intermediate layer of a propylene or polyester type polymer and a paper layer.

The present invention also provides a box-shaped vessel prepared from a laminate film or sheet as defined above.

The present invention additionally provides a process for the preparation of a laminate film or sheet as defined above which comprises extrusion-coating a propylene or polyester-type polymer on the surface of a paper layer to form an intermediate layer and extrusion-coating poly-4-methyl-1-pentene to form an outer layer on the intermediate layer while blowing gas at the outer layer being formed by the extrusion coating.

The present invention further provides a process for producing a box-shaped vessel comprising folding a laminate film or sheet as defined above.

In the film, sheet or vessel of the present invention permeation of oil and fat components from a contained cooked food can be prevented. The carton has excellent oil resistance.

In this laminated film or sheet, the melting point of the poly-4-methyl-1-pentene constituting the inner layer is generally 190 to 245°C and preferably 200 to 235°C. The propylene polymer constituting the intermediate layer is generally a propylene homopolymer of propylene/ethylene random copolymer having MFR (melt flow rate) of 1 to 200 g/10 min, preferably 10 to 100 g/100 min.

In the laminate film or sheet of the present invention, the covering layer is formed of poly-4-methyl-1-pentene.

The poly-4-methyl-1-pentene used in the present invention is a homopolymer of 4-methyl-1-pentene or a copolymer of 4-methyl-1-pentene with up to 20 mole%, preferably up to 12 mole%, of an  $\alpha$ -olefin having 2 to 20 carbon atoms, such as ethylene, propylene, 1-butene, 1-hexene, 1-octene, 1-decene, 2-tetradecene or 1-octadecene. Poly-4-methyl-1-pentene having an ordinary melting point, for example a melting point of 190 to 245°C, may, for instance, be used.

Crystalline poly-4-methyl-1-pentene having a melting point of 190 to 230°C is preferably used. This polymer can be prepared according to a known process, for example, the process disclosed in JP-A-59-206418. The MFR of the poly-4-methyl-1-pentene is preferably 5 to 1000 g/10 min, and poly-4-methyl-1-pentene having an MFR of 70 to 300 g/10 min is especially preferably used because the moldability and heat resistance thereof are very good.

In general, an increase of the amount of the  $\alpha$ -olefin to be copolymerized results in a lowering of the melting point, but the melting point is also influenced by the kind of the  $\alpha$ -olefin, the catalyst used and other polymerization conditions. However, since the relationships between these factors and the melting point can be experimentally determined in advance, a person skilled in the art will easily obtain a poly-4-methyl-1-pentene having a desired melting point.

If a layer of poly-4-methyl-1-pentene having a relatively low melting point of 190 to 230 °C is laminated with a paper layer, even under heating in an electronic oven, the above-mentioned oil and fat components do not permeate into the paper layer, and therefore a laminate film or sheet suitable as a vessel material can be provided.

Papers customarily used as the material for boxes or cases, such as clay-coated paper and milk carton paper, can be used as the paper constituting the paper layer. However, use clay-coated paper is recommended because the permeation rate of oil and fat components can be maintained at a very low level even if pinholes or small cracks are formed in the poly-4-methyl-1-pentene layer by unexpected shock or rubbing or even if the oil and fat components happen to permeate from the end face of the laminated film or sheet. Moreover, since this clay-coated paper has excellent printability, the laminate sheet or film is suitably used as a base material to be printed on when a print layer is formed on the surface of the vessel according to need.

The poly-4-methyl-1-pentene and propylene and polyester type polymers constituting the inner layer (covering let-

ter) and intermediate layer have excellent adaptability to extrusion coating at a high speed, and therefore can be extrusion-coated at a high speed by using an ordinary extrusion-coating apparatus. By this extrusion coating a laminate film or sheet having excellent mechanical properties and interlaminar adhesiveness can be obtained. If a gas such as air or nitrogen gas is blown to both the ends of the extruded film or sheet at the extrusion coating, neck-in or edge wave at 5 both ends of the extruded film or sheet can be prevented, and in this case, if blowing of the gas is effected in the vicinity of the point of contact of the extruded film or sheet with the paper substrate from the side where the extruded film or sheet is not contacted with the paper substrate, the extrusion coating is accomplished at a high speed more stably.

As the method for blowing a gas to the extruded film or sheet, there can be mentioned a method in which a metal tube such as an aluminium tube or copper tube or a conduit tube such as a thermoplastic resin tube is arranged downstream of a die, and a gas is blown to the extruded film or sheet of poly-4-methyl-1-pentene and propylene or polyester-type polymer at an optional point in the course of up to the point of the contact of the film or sheet extruded from the die with the paper substrate, preferably in the vicinity of the contact point where the extruded film or sheet is coated on the paper substrate. The pressure of the gas to be blown is appropriately determined in view of the thickness of the extruded film or sheet to be coated. However, the pressure of the gas is generally 0.5 to 5 kg/cm<sup>2</sup>G. The aperture of the 10 top end of a nozzle for blowing the gas is generally 1 to 50 mm<sup>2</sup> and preferably 5 to 10 mm<sup>2</sup>. The distance between the top end of the nozzle and the extruded film or sheet is generally 2 to 100 mm and preferably 5 to 20 mm. If the top end of the blowing nozzle is directed to the outer side of the extruded film or sheet from the inner side thereof, the film or sheet is expanded by the blown gas and the effect of further narrowing neck-in is preferably attained.

When the poly-4-methyl-1-pentene and propylene or polyester-type polymer is extrusion-coated, the extrusion temperature is ordinarily 250 to 370 °C and preferably 290 to 340 °C. The extrusion-coating speed (the take-up speed of the covering material) is generally at least 100 m/min and preferably 150 to 500 m/min.

The propylene polymer which may constitute the intermediate layer in the present invention includes a crystalline homopolymer having an MFR of 1 to 200 g/10 min, preferably 10 to 100 g/10 min, and a crystalline copolymer of propylene with up to 15 mole% of other olefin such as ethylene, 1-butene, 1-hexene, 4 -methyl-1-pentene or 1-octene.

25 The thicknesses of the poly-4-methyl-1-pentene layer, propylene or polyester type polymer layer and paper layer can be optionally selected, but the thicknesses thereof are generally 1 to 2000 µm, 1 to 2000 µm and 5 to 5000 µm, respectively, and preferably 5 to 50 µm, 5 to 50 µm and 100 to 600 µm, respectively.

Known additives such as a weathering stabilizer, a heat stabilizer, an antistatic agent, an antifogging agent, an anti-blocking agent, a slip agent and a colourant can be incorporated in each of the poly-4-methyl-1-pentene, and propylene 30 and polyester type polymer used in the present invention, so far as the attainment of the intended objects of the present invention is not inhibited.

By adopting a laminate structure of poly-4-methyl-1-pentene layer/propylene or polyester-type polymer layer/paper layer for the laminated film or sheet, even if oil and fat components contact with the poly-4-methyl-1-pentene layer, permeation of these components can be completely prevented. A carton prepared from this laminated film or sheet has 35 excellent oil resistance, and therefore can be advantageously used as a tray to be used for cooking in an electronic oven, a carton for a food containing oil and fat components, such as a cake or a bun, and other packaging vessels.

The polyester type polymer which may constitute the intermediate layer in the present invention is generally a thermoplastic polyester resin comprising main recurring units of ethylene terephthalate or butylene terephthalate and having an intrinsic viscosity ( $\eta_1$ ) of 0.4 to 1.5 dL/g, preferably 0.5 to 1.2 dL/g, and a melting point of 210 to 265 °C, preferably 40 220 to 260 °C, which is derived from terephthalic acid as the main acid component and a diol such as ethylene glycol or 1,4-butane-diol as the main polyhydric alcohol component. Polyethylene terephthalate is preferably used since this polyester has excellent extrusion lamination processability.

The laminated film or sheet of the present embodiment can be prepared by extrusion-coating poly-4-methyl-1-pentene and an intermediate layer-forming polyester directly on paper by co-extrusion. Alternatively, there can be adopted 45 a method in which, in order to further increase the adhesion strength between the paper and intermediate layer, according to a known method, the paper surface is coated with an anchor coat agent of an organic titanium, polyethyleneimine or isocyanate type compound or an adhesive polyethylene or high-pressure polyethylene is coated as the undercoat on the paper surface, and the poly-4-methyl-1-pentene and polyester type polymer are extrusion-coated on the treated paper surface. In the latter case, a laminate film or sheet having a four-layer structure is obtained.

50 The present invention is now further described in the following Examples.

In the Examples, the melting point, MFR and initial modulus of elasticity were measured according to the following methods.

#### Melting Point

55 By using a differential scanning calorimeter (model DSC-II supplied by Perkin-Elmar), a sample was melted at 260 °C for 5 minutes, cooled to room temperature at a rate of 20 °C/min to effect crystallization and maintained at room temperature. An endothermic curve was determined at a temperature-elevating rate of 10 °C/min, and the peak tempera-

ture was designated as the melting point. The poly-4-methyl-1-pentene used in the present invention shows one endothermic peak or a plurality of endothermic peaks. When a plurality of peaks were observed, the highest peak temperature was designated as the melting point.

## 5 MFR

The MFR value was measured under a load of 5 kg at 260 °C according to ASTM D-1238.

## Initial Modulus of Elasticity

10 The initial modulus of elasticity was measured according to ASTM S-790 (the test speed was adjusted to 5 mm/min).

## MFR of Polypropylene

15 The MFR of polypropylene was measured under a load of 2.16 kg at 230 °C according to ASTM D-1238.

Intrinsic Viscosity ( $\eta$ ) of polyethylene Terephthalate Resin

The intrinsic viscosity of a polyethylene terephthalate resin was measured at a temperature of 23°C in a phenol/tetrachloroethane mixed solvent (weight ratio = 1/1).

## 20 Example 1

A crystalline copolymer of 4-methyl-1-pentene with 1-decene (1-decene content of 2.5% by weight) (hereinafter referred to as "PMP (I)") having a melting point of 237°C, MFR of 180 g/10 min and an initial flexural modulus of elasticity of 13000 kg/cm<sup>2</sup>, and a propylene homopolymer (hereinafter referred to as "PP (I)") having an MFR of 30 g/10 min were independently melted in extruders having a screw diameter of 65 mm, and the melts were laminated on a milk carton paper having a base weight of 290 g/mm<sup>2</sup> using a coextrusion two-layer die. The total coating thickness was 30 µm and each of the coating layers had a thickness of 15 µm. A case having a size of 10 cm x 10 cm x 5 cm (depth) was prepared from the obtained laminated paper so that the coating layer was located on the inner surface.

30 Then, weighed 40 g of commercially available margarine (supplied by Yukijirushi Nyugyo) was placed in the case and heated for 30 seconds by an electronic oven (Type NE-A740 supplied by Matsushita Denki Sangyo). After the heating, the case was allowed to stand still at room temperature, and the number of days required for the margarine to exude to the outer surface of the paper layer was determined by visual inspection.

A case prepared in the same manner as described above was charged with a starting powder of a chocolate cake (House Range Gourmet supplied by House Shokuhin) and water, and cooking was carried out for 3 minutes and 30 seconds using the same electronic oven as described above. After the cooking, exudation of the contents to the outer surface was examined without taking out the contents from the case.

35 Similarly, a case prepared in the same manner as described above was charged with a starting powder of a butter cake (House Range Gourmet supplied by House Shokuhin) and water, and cooking was carried out at a set temperature of 190°C for 20 minutes in an oven. After the cooking, exudation of the contents to the outer surface was examined without taking out the contents from the case.

40 The results obtained are shown in Table 2.

## Example 2

45 The procedures of Example 1 were repeated in the same manner except that the coating thickness of PMP (I) used was changed to 10 µm and the coating thickness of PP (I) was changed to 20 µm. The results obtained are shown in Table 2.

## 50 Example 3

The procedures of Example 1 were repeated in the same manner except that an ethylene/propylene random copolymer having ethylene content of 5% by weight (hereinafter referred to as "PP (II)") was used instead of the PP (I). The results obtained are shown in Table 2.

## 55 Comparative Example 1

The procedures of Example 1 were repeated in the same manner except that a resin mixture comprising PMP (I)

and PP (I) in a ratio of 3:1 was used instead of PP (I). The results obtained are shown in Table 2.

Comparative Example 2

5        The procedures of Example 1 were repeated in the same manner except that a resin mixture comprising PMP (I) and PP (I) in a ratio of 1:1 was used instead of PP (I). A product having a total coating thickness of 30 µm was obtained.

      The exudation test was carried out in the same manner as described in Example 1. The results obtained are shown in Table 2.

10      Comparative Example 3

      By using a single-layer die instead of the coextrusion two-layer die used in Example 1, PMP (I) was laminated in a coating thickness of 15 µm. Then, PMP (I) was laminated again in a thickness of 15 µm while opening a roll of the semi-finished product once wound to obtain a product having a total coating thickness of 30 µm. This product was also subjected to the exudation test in the same manner as described in Example 1. The results obtained are shown in Table 1.

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Table 1

<u>Structure</u>	<u>Example 1</u>	<u>Example 2</u>	<u>Example 3</u>	<u>Comparative Example 1</u>	<u>Comparative Example 2</u>	<u>Comparative Example 3</u>
inner layer	PMP(I)	PMP(I)	PMP(I)	PMP(I)	PMP(I)	PMP(I)
intermediate layer	PP(I)	PP(I)	PP(III)	PMP(I)+PP(I) (75) (25)	PMP(I)+PP(I) (50) (50)	PMP(I)
outer layer	milk carton paper	milk carton paper	milk carton paper	milk carton paper	milk carton paper	milk carton paper
<u>Thickness (μm)</u>						
inner layer	15	10	15	15	15	15
intermediate layer	15	20	15	15	15	15
outer layer	300	300	300	300	300	300
Number of days for exudation of margarine	exudation did not occur for 20 days, and test was stopped	same as in Example 1	same as in Example 1	1	2	4
Number of days for exudation of chocolate cake	ditto	same as in Example 1	same as in Example 1	3	3	3
Number of days for exudation of butter cake	ditto	same as in Example 1	same as in Example 1	5	5	5

Note Each parenthesized value indicates the blending ratio.

Example 4

5 PMP (I) and polyethylene terephthalate (hereinafter referred to as "PET (I)") having an intrinsic viscosity ( $\eta$ ) of 0.6 were independently melted in extruders having a screw diameter of 65 mm and the melts were laminated on milk carton paper having a base weight of 290 g/mm<sup>2</sup> using a coextrusion two-layer die. The total coating thickness was 30  $\mu\text{m}$ , and each of the laminated layers had a thickness of 15  $\mu\text{m}$ . A case having a size of 10 cm x 10 cm x 5 cm (depth) was prepared from the obtained laminated paper so that the coating layer was located on the inner surface.

10 Then, weighed 40 g of commercially available margarine (supplied by Yukijirushi Nyugyo) was placed in the case and heated for 30 seconds by an electronic oven (Type NE-A740 supplied by Matsushita Denki Sangyo). After the heating, the case was allowed to stand still at room temperature, and the number of days required for the margarine to exude to the outer surface of the paper layer was determined by visual inspection.

15 A case prepared in the same manner as described above was charged with a starting powder of a chocolate cake (House Range Gourmet supplied by House Shokuhin) and water, and cooking was carried out for 3 minutes and 30 seconds using the same electronic oven as described above. After the cooking, exudation of the contents to the outer surface was examined without taking out the contents from the case.

20 Similarly, a case prepared in the same manner as described above was charged with a starting powder of a butter cake (House Range Gourmet supplied by House Shokuhin) and water, and cooking was carried out at a set temperature of 190°C for 20 minutes in an oven. After the cooking, exudation of the contents to the outer surface was examined without taking out the contents from the case.

25 The results obtained are shown in Table 2.

Example 5

The procedures of Example 6 were repeated in the same manner except that the coating thickness of PMP (I) was changed to 20  $\mu\text{m}$  and the thickness of the coating layer of PET (I) was changed to 10  $\mu\text{m}$ . The results obtained are shown in Table 2.

Table 2

30 Structure	Example 4	Example 5
inner layer	PMP(I)(15)	PMP(I)(20)
intermediate layer	PET(I) (15)	PET(I) (10)
outer layer	milk carton paper (300)	milk carton paper (300)
Number of days for exudation of margarine	exudation did not occur for 20 days, and test was stopped	same as in Example 4
Number of days for exudation of chocolate cake (electronic oven)	ditto	same as in Example 4
Number of days for exudation of butter cake (oven)	ditto	same as in Example 4

45 Note Unit of each parenthesized value is  $\mu\text{m}$ .

## Claims

1. A laminate film or sheet having at least three layers comprising a layer of poly-4-methyl-1-pentene having a melting point of 190°C to 245°C, an intermediate layer of a propylene or polyester type polymer and a paper layer.
2. A film or sheet according to claim 1 wherein the poly-4-methyl-1-pentene has a melting point of 190 to 230°C.
3. A film or sheet according to claim 1 or 2 wherein the thickness of the poly-4-methyl-1-pentene layer is 1 to 2000  $\mu\text{m}$  and the thickness of the paper layer is 5 to 5000  $\mu\text{m}$ .
4. A film or sheet according to any one of claims 1 to 3 wherein the melt flow rate of the poly-4-methyl-1-pentene is 5 to 1000 g/10 min.

5. A film or sheet according to any one of claims 1 to 4 wherein the intermediate layer is composed of polypropylene having a melt flow rate of 1 to 200 g/10 min.
6. A film or sheet according to any one of claims 1 to 7 wherein the polypropylene layer has a thickness of 5 to 5000  
5 µm.
7. A film or sheet according to claim 5 wherein the intermediate layer is composed of polyethylene terephthalate having an intrinsic viscosity ( $\zeta$ ) of 0.4 to 1.5 dL/g and a melting point of 210 to 265°C.
- 10 8. A box-shaped vessel prepared from a laminate film or sheet according to any one of claims 1 to 7.
9. A process for the preparation of a laminate film or sheet as defined in any one of claims 1 to 7, which comprises extrusion-coating a propylene or polyester-type polymer on the surface of a paper layer to form an intermediate layer and extrusion-coating poly-4-methyl-1-pentene to form an outer layer on the intermediate layer while blowing  
15 gas at the outer layer being formed by the extrusion coating.
10. A process for producing a box-shaped vessel comprising folding a laminate film or sheet according to any one of claims 1 to 7.

20 **Patentansprüche**

1. Laminatfolie oder -bahn mit mindestens drei Schichten, umfassend eine Schicht aus Poly-4-methyl-1-penten mit einem Schmelzpunkt von 190 bis 245°C, eine Zwischenschicht aus einem Polymer vom Propylen- oder Polyester-  
25 typ und eine Papierschicht.
2. Folie oder Bahn nach Anspruch 1, wobei das Poly-4-methyl-1-penten einen Schmelzpunkt von 190 bis 230°C hat.
3. Folie oder Bahn nach Anspruch 1 oder 2, wobei die Dicke der Poly-4-methyl-1-penten-Schicht 1 bis 2000 µm und die Dicke der Papierschicht 5 bis 5000 µm beträgt.
- 30 4. Folie oder Bahn nach einem der Ansprüche 1 bis 3, wobei die Fließfähigkeit des Poly-4-methyl-1-pentens 5 bis 1000 g/10 min beträgt.
5. Folie oder Bahn nach einem der Ansprüche 1 bis 4, wobei die Zwischenschicht aus Polypropylen mit einer Fließ-  
35 fähigkeit von 1 bis 200 g/10 min besteht.
6. Folie oder Bahn nach einem der Ansprüche 1 bis 7, wobei die Polypropylenschicht eine Dicke von 5 bis 5000 µm hat.
- 40 7. Folie oder Bahn nach Anspruch 5, wobei die Zwischenschicht aus Polyethylenterephthalat mit einer Grundviskosität ( $\eta$ ) von 0,4 bis 1,5 dL/g und einem Schmelzpunkt von 210 bis 265°C besteht.
8. Kastenförmiges Gefäß, hergestellt aus einer Laminatfolie oder -bahn nach einem der Ansprüche 1 bis 7.
- 45 9. Verfahren zur Herstellung einer Laminatfolie oder -bahn nach einem der Ansprüche 1 bis 7, umfassend das Extrusionsbeschichten der Oberfläche einer Papierschicht mit einem Polymer vom Propylen- oder Polyester-  
typ zur Bildung einer Zwischenschicht und Extrusionsbeschichten der Zwischenschicht mit Poly-4-methyl-1-penten zur Bildung einer Außenschicht, wobei Gas auf die beim Extrusionsbeschichten entstandene Außenschicht aufgeblasen wird.
- 50 10. Verfahren zur Herstellung eines kastenförmigen Gefäßes, umfassend das Falten einer Laminatfolie oder -bahn nach einem der Ansprüche 1 bis 7.

55 **Revendications**

1. Film ou feuille stratifiés comportant au moins trois couches, comprenant une couche de poly(4-méthyl-1-pentène) ayant un point de fusion compris entre 190 et 245 °C, une couche intermédiaire en un polymère de type polypropylène ou polyester et une couche de papier.

2. Film ou feuille selon la revendication 1 dans lesquels le poly(4-méthyl-1-pentène) a un point de fusion compris dans l'intervalle allant de 190 à 230 °C.
3. Film ou feuille selon la revendication 1 ou 2 dans lesquels l'épaisseur de la couche de poly(4-méthyl-1-pentène) est comprise entre 1 et 2000 µm et l'épaisseur de la couche de papier est comprise entre 5 et 5000 µm.
4. Film ou feuille selon l'une quelconque des revendications 1 à 3 dans lesquels l'indice de fluidité à chaud du poly(4-méthyl-1-pentène) est compris entre 5 et 1000 g/10 min.
- 10 5. Film ou feuille selon l'une quelconque des revendications 1 à 4 dans lesquels la couche intermédiaire est composée de polypropylène ayant un indice de fluidité à chaud compris entre 1 et 200 g/10 minutes.
6. Film ou feuille selon l'une quelconque des revendications 1 à 7 dans lesquels la couche de polypropylène a une épaisseur de 5 à 5000 µm.
- 15 7. Film ou feuille selon la revendication 5 dans lesquels la couche intermédiaire est constituée de poly(éthylène téraphthalate) ayant une viscosité intrinsèque ( $\zeta$ ) de 0,4 à 1,5 dl/g et un point de fusion compris entre 210 et 265 °C.
8. Récipient ayant la forme d'une boîte préparé à partir d'une feuille stratifiée ou d'un film stratifié selon l'une quelconque des revendications 1 à 7.
- 20 9. Procédé de préparation d'un film ou d'une feuille stratifiés définis dans l'une quelconque des revendications 1 à 7, qui comprend les étapes consistant à déposer par extrusion un polymère de type polypropylène ou polyester à la surface d'une couche de papier de manière à former une couche intermédiaire, puis à déposer par extrusion du poly(4-méthyl-1-pentène) pour former une couche extérieure sur la couche intermédiaire tout en soufflant un gaz sur la couche extérieure en cours de formation par extrusion-revêtement.
10. Procédé de préparation d'un récipient ayant la forme d'une boîte comprenant le pliage d'un film ou d'une feuille stratifiés selon l'une quelconque des revendications 1 à 7.

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